UNITED STATES PATENT OFFICE.

HEINRICH JORDAN AND WILHELM NEELMEIER, OF LEVERKUSEN, NEAR COLOGNE, GERMANY, ASSIGNORS TO FAR ENFABRIKEN VORM. FRIEDR. BAYER & CO., OF ELBERFELD, GERMANY, A CORPORATION OF GERMANY.

BLUE DISAZO DYE.

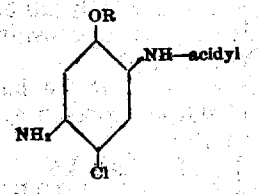
1,000,269. Specification of Letters Patent. Patented Aug. 8, 1911.

No Drawing. Application filed May 10, 1911. Serial No. 626,245.

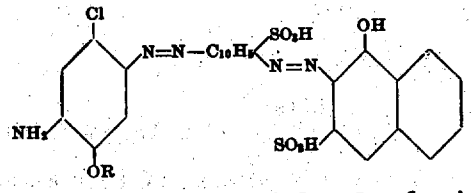
To all whom it may concern:

Be it known that we, HEINRICH JORDAN and WILHELM NEELMEIER, doctors of philosophy, chemists, citizens of the German Empire, residing at Leverkusen, near Cologne, Germany, have invented new and useful Improvements in Blue Disazo Dye, of which the following is a specification.

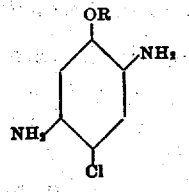
It has been found that valuable disazodyestuffs which can be diazotized and developed on the fiber are obtained by combining the diazo compounds of 5-amino-4-chloro-2-acidylamino-1-phenol ethers of the formula:



(R means alkyl) with 1-naphthylamin-6- or 7-sulfonic acid or with a mixture of both acids, rediazotizing the compounds thus obtained, combining them with 1.3-naphthol sulfonic acid and splitting off the acidyl radical. Blue cotton dyestuffs are thus obtained of the formula:

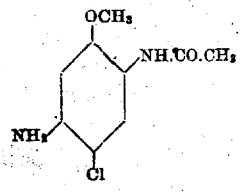


They are after being dried and pulverized dark powders soluble in water generally with a blue coloration. Upon treatment with stannous chlorid and hydrochloric acid they are split up, a 2.5-diaminobenzene derivative of the formula:

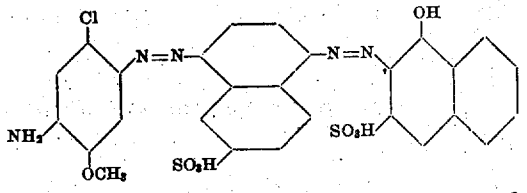


1.4-naphthylenediamin sulfonic acid and 2-amino-1-naphthol-3-sulfonic acid are obtained. They can be diazotized on the fiber and developed *e. g.* with beta-naphthol the result being bright greenish-blue shades of excellent fastness to washing which can be discharged with hydro-sulfite to a pure white.

In order to illustrate the new process more fully the following example is given, the parts being by weight:—215 parts of 5-amino-4-chloro-2-acetylamino-1-anisol of the formula:



are well mixed with 7000 parts of water, 250 parts of hydrochloric acid (19° Bé.) are added and the mixture is diazotized at 10° C. with 69 parts of sodium nitrite. The diazo compound thus obtained is then allowed to run into a solution of 245 parts of the sodium salt of 1-naphthylamin-6-sulfonic acid to which 250 parts of sodium acetate (100 per cent.) have been added. The combination is finished after a few minutes. Hylrochloric acid is added until the free mineral acid is present and the aminoazo dyestuff is rediazotized at 10° C. with 69 parts of sodium nitrite. After about one hour the diazotation is complete. The diazo compound is then allowed to run into a solution of 246 parts of the sodium salt of 1-naphthol-3-sulfonic acid in water containing 700 parts of soda. The dyestuff which forms is filtered off, the paste is then dissolved in 7000 parts of hot water and the dyestuff is saponified by boiling it for ¼ hour with 1400 parts of soda lye (30 per cent.). The hot solution is immediately cooled with cold water, the excess of the soda lye is neutralized and the dyestuff is filtered off. It is after being dried and pulverized a dark powder of the formula:

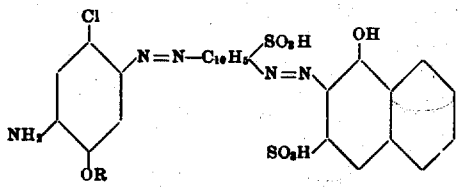


soluble in water with a reddish-blue coloration, soluble in concentrated sulfuric acid with a green coloration and yielding

upon reduction with stannous chlorid and hydrochloric acid 2.5-diamino-4-chloro-1-anisol, 1.4-naphthylenediamin-6-sulfonic acid and 1-naphthol-2-amino-3-sulfonic acid. It dyes cotton in blue shades, which when diazotized on the fiber and developed with beta-naphthol change into a greenish-blue of excellent fastness to washing and to light. It can be discharged to a pure white with hydrosulfite. The same method is employed on using the corresponding phenethols or the 1.7-naphthylamin sulfonic acid.

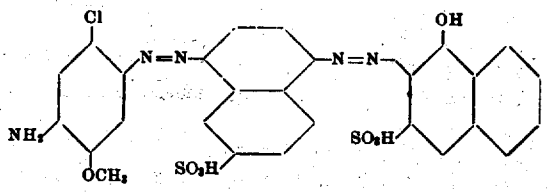
We claim:—

1. The herein described disazodyestuffs of the formula:



which dyestuffs are after being dried and pulverized dark powders soluble in water generally with a blue coloration; yielding upon treatment with stannous chlorid and hydrochloric acid a 2.5-diaminobenzene derivative, 1.4-naphthylenediamin sulfonic acid and 2-amino-1-naphthol-3-sulfonic acid; which dyes can be diazotized on the fiber and developed with beta-naphthol giving bright greenish-blue shades of excellent fastness to washing which can be discharged with hydrosulfite to a pure white, substantially as described.

2. The herein described disazodyestuff of the formula



which dyestuff is after being dried and pulverized a dark powder soluble in water with a reddish-blue coloration; soluble in concentrated sulfuric acid with a green coloration; yielding upon treatment with stannous chlorid and hydrochloric acid 2.5-diamino-4-chloro-1-anisol, 1.4-naphthylenediamin-6-sulfonic acid and 1-naphthol-2-amino-3-sulfonic acid; dyeing cotton in blue shades, which when diazotized on the fiber and developed with beta-naphthol change into a greenish-blue which can be discharged to a pure white with hydrosulfite, substantially as described.

In testimony whereof we have hereunto set our hands in the presence of two subscribing witnesses.

HEINRICH JORDAN. [L. S.]
WILHELM NEELMEIER. [L. S.]

Witnesses:
CHAS. J. WRIGHT,
ALFRED HENKEL.

Corrections in Letters Patent No. 1,000,269.

It is hereby certified that in Letters Patent No. 1,000,269, granted August 8, 1911, upon the application of Heinrich Jordan and Wilhelm Neelmeier, of Leverkusen, near Cologne, Germany, for an improvement in "Blue Disazo Dye," errors appear in the printed specification requiring correction as follows: Page 1, line 80, for the word "Hylrochloric" read *Hydrochloric;* page 2, line 28, for the word "hlydrochloric" read *hydrochloric;* and that the said Letters Patent should be read with these corrections therein that the same may conform to the record of the case in the Patent Office.

Signed and sealed this 3rd day of October, A. D., 1911.

[SEAL.]

E. B. MOORE,
Commissioner of Patents.

upon reduction with stannous chlorid and hydrochloric acid 2.5-diamino-4-chloro-1-anisol, 1.4-naphthylenediamin-6-sulfonic acid and 1-naphthol-2-amino-3-sulfonic acid. It dyes cotton in blue shades, which when diazotized on the fiber and developed with beta-naphthol change into a greenish-blue of excellent fastness to washing and to light. It can be discharged to a pure white with hydrosulfite. The same method is employed on using the corresponding phenethols or the 1.7-naphthylamin sulfonic acid.

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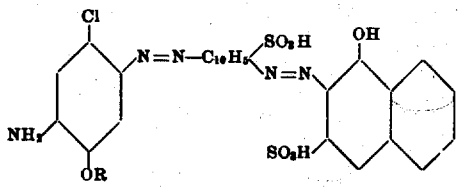

which dyestuffs are after being dried and pulverized dark powders soluble in water generally with a blue coloration; yielding upon treatment with stannous chlorid and hydrochloric acid a 2.5-diaminobenzene derivative, 1.4-naphthylenediamin sulfonic acid and 2-amino-1-naphthol-3-sulfonic acid; which dyes can be diazotized on the fiber and developed with beta-naphthol giving bright greenish-blue shades of excellent fastness to washing which can be discharged with hydrosulfite to a pure white, substantially as described.

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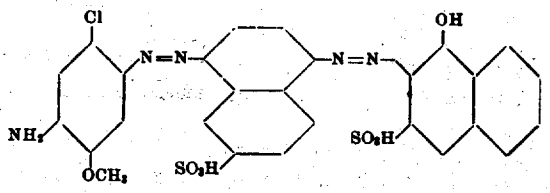

which dyestuff is after being dried and pulverized a dark powder soluble in water with a reddish-blue coloration; soluble in concentrated sulfuric acid with a green coloration; yielding upon treatment with stannous chlorid and hydrochloric acid 2.5-diamino-4-chloro-1-anisol, 1.4-naphthylenediamin-6-sulfonic acid and 1-naphthol-2-amino-3-sulfonic acid; dyeing cotton in blue shades, which when diazotized on the fiber and developed with beta-naphthol change into a greenish-blue which can be discharged to a pure white with hydrosulfite, substantially as described.

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